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5-Iodo-7-methyl-2-(4-methylphenyl)-3-methylsulfinyl-1-benzofuran

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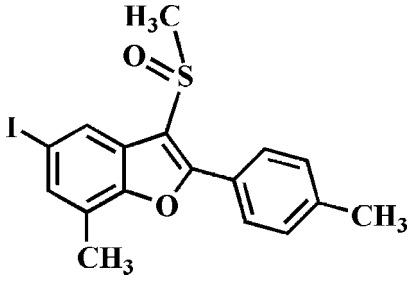
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.017; wR factor = 0.044; data-to-parameter ratio = 20.0.

In the title compound, $C_{17}H_{15}IO_2S$, the dihedral angle between the benzofuran group (r.m.s. deviation = 0.009 Å) and the 4-methylbenzene ring is 12.69 (5)°. In the crystal, molecules are linked *via* pairs of I···O [I···O = 3.164 (1) Å, C—I···O = 166.63 (5)°] contacts into inversion-related dimers.

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2008, 2010). For a review of halogen bonding, see: Politzer *et al.* (2007).



Experimental

Crystal data

$C_{17}H_{15}IO_2S$
M_r = 410.25
Triclinic, $P\overline{1}$
a = 7.6816 (3) Å
b = 9.6478 (4) Å
c = 11.3738 (4) Å
α = 75.409 (2)°
β = 84.062 (2)°
γ = 71.684 (2)°
V = 774.14 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.20 mm^{-1}
T = 173 K
0.25 × 0.25 × 0.21 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.607, T_{max} = 0.652
14337 measured reflections
3858 independent reflections
3637 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.044
S = 1.09
3858 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: PK2514).

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supporting information

Acta Cryst. (2014). E**70**, o178 [doi:10.1107/S1600536814000865]

5-Iodo-7-methyl-2-(4-methylphenyl)-3-methylsulfinyl-1-benzofuran

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S1. Comment

As a part of our continuing study of 5-iodo-7-methyl-3-methylsulfinyl-1-benzofuran derivatives containing phenyl (Choi *et al.*, 2008) and 4-fluorophenyl (Choi *et al.*, 2010) substituents in 2-position, we report here the crystal structure of the title compound.

In the title molecule (Fig. 1), the benzofuran unit is essentially planar, with a mean deviation of 0.009 (1) Å from the least-squares plane defined by the nine constituent atoms. The 4-methylphenyl ring is essentially planar, with a mean deviation of 0.005 (1) Å from the least-squares plane defined by the six constituent atoms. The dihedral angle formed by the benzofuran ring system and the 4-methylphenyl ring is 12.69 (5)°. In the crystal structure, molecules are connected by pairs of I···O halogen-bonds between the iodine atom and the O atom of the S═O unit [I1···O2^{i} = 3.164 (1) Å, C4—I1···O2^{i} = 166.63 (5)°, Symmetry code i = - *x*, - *y* + 1, - *z*.] (Politzer *et al.*, 2007), forming inversion-related dimers.

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 224 mg, 1.0 mmol) was added in small portions to a stirred solution of 5-iodo-7-methyl-2-(4-methylphenyl)-3-methylsulfanyl-1-benzofuran (355 mg, 0.9 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 4h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated at reduced pressure. The residue was purified by column chromatography (hexane–ethyl acetate, 1:1 v/v) to afford the title compound as a colorless solid [yield 73%, m.p. 483–484 K; R_f = 0.48 (hexane–ethyl acetate, 1:1 v/v)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in acetone at room temperature.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å for aryl and 0.98 Å for methyl H atoms. U_{iso}(H) = 1.2U_{eq} (C) for aryl and 1.5U_{eq}(C) for methyl H atoms. The positions of methyl hydrogens were optimized rotationally.

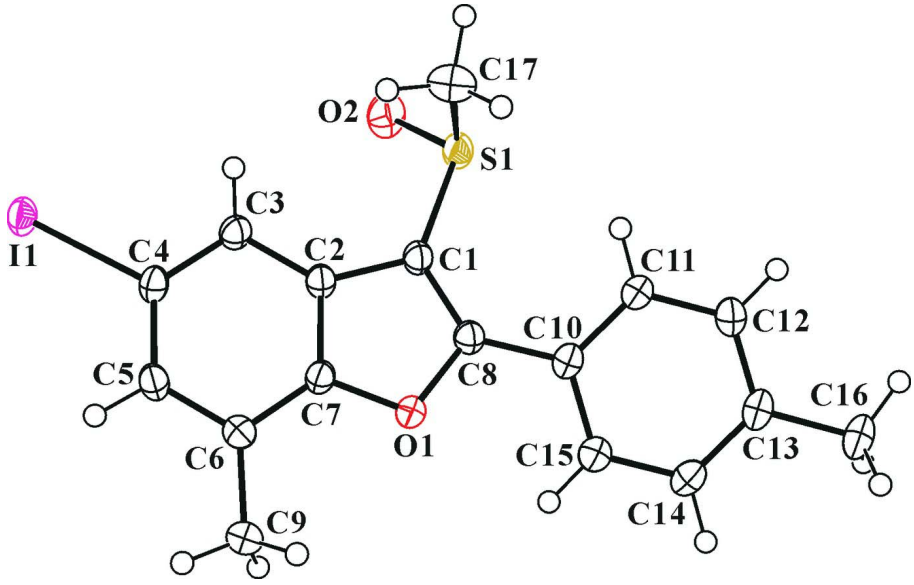

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

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$C_{17}H_{15}IO_2S$
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$a = 7.6816\ (3)$ Å
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$c = 11.3738\ (4)$ Å
$\alpha = 75.409\ (2)°$
$\beta = 84.062\ (2)°$
$\gamma = 71.684\ (2)°$
$V = 774.14\ (5)$ Å^3
$Z = 2$
$F(000) = 404$
$D_x = 1.760$ Mg m^{-3}
Melting point = 483–484 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9908 reflections
$\theta = 2.6–28.4°$
$\mu = 2.20$ mm^{-1}
$T = 173$ K
Block, colourless
0.25 × 0.25 × 0.21 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: rotating anode
Graphite multilayer monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.607$, $T_{max} = 0.652$
14337 measured reflections
3858 independent reflections
3637 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 28.4°$, $\theta_{min} = 1.9°$
$h = -10\rightarrow10$
$k = -12\rightarrow12$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.017$
$wR(F^2) = 0.044$
$S = 1.09$
3858 reflections
193 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0222P)^2 + 0.2874P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	−0.019314 (14)	0.745216 (11)	−0.052801 (9)	0.02619 (4)
S1	0.34602 (7)	0.20502 (4)	0.38345 (4)	0.03019 (9)
O1	0.19544 (15)	0.59238 (12)	0.48325 (10)	0.0235 (2)
O2	0.2168 (2)	0.19491 (16)	0.29934 (14)	0.0444 (4)
C1	0.2754 (2)	0.39157 (17)	0.40042 (15)	0.0230 (3)
C2	0.1875 (2)	0.52209 (17)	0.30798 (14)	0.0216 (3)
C4	0.0549 (2)	0.69626 (18)	0.12932 (15)	0.0228 (3)
C5	0.0068 (2)	0.81375 (18)	0.18956 (15)	0.0247 (3)
H5	−0.0561	0.9125	0.1469	0.030*
C6	0.0492 (2)	0.78875 (18)	0.31048 (15)	0.0242 (3)
C7	0.1397 (2)	0.64097 (17)	0.36481 (14)	0.0217 (3)
C8	0.2778 (2)	0.43936 (17)	0.50377 (14)	0.0221 (3)
C9	0.0053 (3)	0.9109 (2)	0.37826 (18)	0.0347 (4)
H9A	−0.1026	0.9073	0.4317	0.052*
H9B	−0.0203	1.0086	0.3202	0.052*
H9C	0.1100	0.8965	0.4272	0.052*
C10	0.3492 (2)	0.36861 (18)	0.62510 (14)	0.0226 (3)
C11	0.4716 (2)	0.22337 (19)	0.65298 (15)	0.0273 (3)
H11	0.5087	0.1680	0.5918	0.033*
C12	0.5393 (2)	0.1594 (2)	0.76906 (16)	0.0282 (3)
H12	0.6204	0.0599	0.7867	0.034*
C13	0.4905 (2)	0.2385 (2)	0.85977 (15)	0.0258 (3)
C14	0.3695 (2)	0.3839 (2)	0.83131 (16)	0.0284 (3)
H14	0.3353	0.4401	0.8921	0.034*
C15	0.2985 (2)	0.44779 (19)	0.71653 (15)	0.0266 (3)

H15	0.2147	0.5462	0.6998	0.032*
C3	0.1452 (2)	0.54937 (18)	0.18552 (15)	0.0237 (3)
H3	0.1771	0.4711	0.1434	0.028*
C16	0.5645 (3)	0.1710 (2)	0.98538 (16)	0.0323 (4)
H16A	0.4648	0.1543	1.0428	0.049*
H16B	0.6178	0.2396	1.0090	0.049*
H16C	0.6592	0.0750	0.9863	0.049*
C17	0.5538 (3)	0.2117 (3)	0.2978 (2)	0.0455 (5)
H17A	0.6066	0.1191	0.2693	0.068*
H17B	0.6414	0.2219	0.3495	0.068*
H17C	0.5270	0.2980	0.2279	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03146 (6)	0.02535 (6)	0.02107 (6)	−0.00844 (4)	−0.00869 (4)	−0.00105 (4)
S1	0.0457 (2)	0.01849 (18)	0.0264 (2)	−0.00770 (17)	−0.01246 (18)	−0.00318 (16)
O1	0.0287 (6)	0.0218 (5)	0.0188 (5)	−0.0053 (4)	−0.0034 (4)	−0.0041 (4)
O2	0.0641 (9)	0.0315 (7)	0.0452 (8)	−0.0174 (7)	−0.0248 (7)	−0.0091 (6)
C1	0.0282 (7)	0.0197 (7)	0.0211 (7)	−0.0075 (6)	−0.0051 (6)	−0.0023 (6)
C2	0.0237 (7)	0.0200 (7)	0.0211 (7)	−0.0076 (6)	−0.0041 (6)	−0.0021 (6)
C4	0.0249 (7)	0.0245 (7)	0.0196 (7)	−0.0097 (6)	−0.0058 (6)	−0.0013 (6)
C5	0.0260 (7)	0.0207 (7)	0.0246 (8)	−0.0053 (6)	−0.0059 (6)	−0.0003 (6)
C6	0.0253 (7)	0.0216 (7)	0.0246 (8)	−0.0053 (6)	−0.0021 (6)	−0.0052 (6)
C7	0.0235 (7)	0.0232 (7)	0.0184 (7)	−0.0071 (6)	−0.0035 (6)	−0.0034 (6)
C8	0.0233 (7)	0.0213 (7)	0.0213 (7)	−0.0070 (6)	−0.0020 (6)	−0.0032 (6)
C9	0.0448 (10)	0.0246 (8)	0.0299 (9)	0.0004 (7)	−0.0061 (8)	−0.0096 (7)
C10	0.0246 (7)	0.0253 (7)	0.0193 (7)	−0.0106 (6)	−0.0024 (6)	−0.0027 (6)
C11	0.0322 (8)	0.0274 (8)	0.0211 (8)	−0.0055 (7)	−0.0045 (7)	−0.0064 (7)
C12	0.0294 (8)	0.0272 (8)	0.0243 (8)	−0.0053 (6)	−0.0058 (7)	−0.0012 (7)
C13	0.0249 (7)	0.0334 (8)	0.0198 (7)	−0.0129 (7)	−0.0040 (6)	−0.0007 (7)
C14	0.0331 (8)	0.0330 (9)	0.0210 (8)	−0.0109 (7)	−0.0013 (7)	−0.0080 (7)
C15	0.0294 (8)	0.0263 (8)	0.0233 (8)	−0.0072 (6)	−0.0024 (6)	−0.0054 (7)
C3	0.0280 (8)	0.0229 (7)	0.0224 (8)	−0.0095 (6)	−0.0048 (6)	−0.0052 (6)
C16	0.0316 (9)	0.0440 (10)	0.0211 (8)	−0.0130 (8)	−0.0071 (7)	−0.0023 (7)
C17	0.0470 (12)	0.0409 (11)	0.0454 (12)	−0.0025 (9)	0.0023 (10)	−0.0194 (10)

Geometric parameters (Å, º)

I1—C4	2.1025 (16)	C9—H9C	0.9800
S1—O2	1.4879 (14)	C10—C15	1.395 (2)
I1—O2^{i}	3.1642 (14)	C10—C11	1.399 (2)
S1—C1	1.7637 (16)	C11—C12	1.387 (2)
S1—C17	1.792 (2)	C11—H11	0.9500
O1—C7	1.3759 (18)	C12—C13	1.387 (2)
O1—C8	1.3799 (18)	C12—H12	0.9500
C1—C8	1.370 (2)	C13—C14	1.396 (3)
C1—C2	1.447 (2)	C13—C16	1.502 (2)

C2—C7	1.389 (2)	C14—C15	1.383 (2)
C2—C3	1.403 (2)	C14—H14	0.9500
C4—C3	1.384 (2)	C15—H15	0.9500
C4—C5	1.402 (2)	C3—H3	0.9500
C5—C6	1.390 (2)	C16—H16A	0.9800
C5—H5	0.9500	C16—H16B	0.9800
C6—C7	1.386 (2)	C16—H16C	0.9800
C6—C9	1.500 (2)	C17—H17A	0.9800
C8—C10	1.455 (2)	C17—H17B	0.9800
C9—H9A	0.9800	C17—H17C	0.9800
C9—H9B	0.9800		

C4—I1—O2^{i}	166.63 (5)	C15—C10—C8	119.69 (15)
O2—S1—C1	107.08 (8)	C11—C10—C8	121.93 (14)
O2—S1—C17	106.95 (10)	C12—C11—C10	120.71 (15)
C1—S1—C17	97.23 (9)	C12—C11—H11	119.6
C7—O1—C8	107.01 (12)	C10—C11—H11	119.6
C8—C1—C2	107.37 (14)	C11—C12—C13	120.98 (16)
C8—C1—S1	127.41 (13)	C11—C12—H12	119.5
C2—C1—S1	125.00 (12)	C13—C12—H12	119.5
C7—C2—C3	119.35 (14)	C12—C13—C14	118.12 (15)
C7—C2—C1	105.00 (14)	C12—C13—C16	121.45 (16)
C3—C2—C1	135.65 (14)	C14—C13—C16	120.44 (15)
C3—C4—C5	122.76 (15)	C15—C14—C13	121.40 (16)
C3—C4—I1	118.82 (11)	C15—C14—H14	119.3
C5—C4—I1	118.40 (12)	C13—C14—H14	119.3
C6—C5—C4	121.36 (15)	C14—C15—C10	120.41 (16)
C6—C5—H5	119.3	C14—C15—H15	119.8
C4—C5—H5	119.3	C10—C15—H15	119.8
C7—C6—C5	114.86 (14)	C4—C3—C2	116.55 (14)
C7—C6—C9	121.60 (15)	C4—C3—H3	121.7
C5—C6—C9	123.53 (15)	C2—C3—H3	121.7
O1—C7—C6	124.21 (14)	C13—C16—H16A	109.5
O1—C7—C2	110.68 (13)	C13—C16—H16B	109.5
C6—C7—C2	125.11 (15)	H16A—C16—H16B	109.5
C1—C8—O1	109.92 (14)	C13—C16—H16C	109.5
C1—C8—C10	135.43 (15)	H16A—C16—H16C	109.5
O1—C8—C10	114.62 (13)	H16B—C16—H16C	109.5
C6—C9—H9A	109.5	S1—C17—H17A	109.5
C6—C9—H9B	109.5	S1—C17—H17B	109.5
H9A—C9—H9B	109.5	H17A—C17—H17B	109.5
C6—C9—H9C	109.5	S1—C17—H17C	109.5
H9A—C9—H9C	109.5	H17A—C17—H17C	109.5
H9B—C9—H9C	109.5	H17B—C17—H17C	109.5
C15—C10—C11	118.36 (15)		

O2—S1—C1—C8	142.69 (15)	S1—C1—C8—O1	−174.32 (11)
C17—S1—C1—C8	−107.04 (17)	C2—C1—C8—C10	−177.62 (17)

O2—S1—C1—C2	−31.25 (17)	S1—C1—C8—C10	7.6 (3)
C17—S1—C1—C2	79.02 (16)	C7—O1—C8—C1	0.29 (17)
C8—C1—C2—C7	−1.04 (17)	C7—O1—C8—C10	178.82 (13)
S1—C1—C2—C7	173.92 (12)	C1—C8—C10—C15	−168.65 (18)
C8—C1—C2—C3	179.06 (17)	O1—C8—C10—C15	13.3 (2)
S1—C1—C2—C3	−6.0 (3)	C1—C8—C10—C11	12.8 (3)
C3—C4—C5—C6	0.5 (3)	O1—C8—C10—C11	−165.23 (14)
I1—C4—C5—C6	179.04 (12)	C15—C10—C11—C12	0.6 (2)
C4—C5—C6—C7	−0.5 (2)	C8—C10—C11—C12	179.16 (15)
C4—C5—C6—C9	178.40 (17)	C10—C11—C12—C13	−1.2 (3)
C8—O1—C7—C6	179.35 (15)	C11—C12—C13—C14	0.7 (2)
C8—O1—C7—C2	−0.99 (17)	C11—C12—C13—C16	−179.36 (16)
C5—C6—C7—O1	179.41 (14)	C12—C13—C14—C15	0.5 (2)
C9—C6—C7—O1	0.5 (3)	C16—C13—C14—C15	−179.46 (16)
C5—C6—C7—C2	−0.2 (2)	C13—C14—C15—C10	−1.1 (3)
C9—C6—C7—C2	−179.08 (16)	C11—C10—C15—C14	0.6 (2)
C3—C2—C7—O1	−178.83 (13)	C8—C10—C15—C14	−178.03 (15)
C1—C2—C7—O1	1.25 (17)	C5—C4—C3—C2	0.1 (2)
C3—C2—C7—C6	0.8 (2)	I1—C4—C3—C2	−178.41 (11)
C1—C2—C7—C6	−179.09 (15)	C7—C2—C3—C4	−0.7 (2)
C2—C1—C8—O1	0.47 (18)	C1—C2—C3—C4	179.15 (17)

Symmetry code: (i) $-x, -y+1, -z$.